National Institute of Standards & Technology

# Certificate of Analysis

## Standard Reference Material® 1974b

### Organics in Mussel Tissue (*Mytilus edulis*)

Standard Reference Material (SRM) 1974b is a frozen mussel tissue homogenate intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, and chlorinated pesticides in marine bivalve mollusk tissue and similar matrices. All of the constituents for which certified and reference values are provided in SRM 1974b were naturally present in the tissue material before processing. A unit of SRM 1974b consists of five bottles each containing approximately 8 g to 10 g (wet basis) of frozen tissue homogenate.

**Certified Concentration Values:** Certified values for concentrations, expressed as mass fractions, for 22 PAHs, 31 PCB congeners, and 7 chlorinated pesticides are provided in Tables 1 to 3. The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on the agreement of results obtained at NIST from two or more chemically independent analytical techniques along with results from an interlaboratory comparison study [1,2]. A certified value for the concentration of total mercury, based on results from NIST and collaborating laboratories, is provided in Table 4. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST.

**Reference Concentration Values:** Reference values for concentrations, expressed as mass fractions, are provided for 16 additional PAHs (some in combination), 8 additional PCB congeners plus total PCBs, 6 additional chlorinated pesticides, total extractable organics (TEO), methylmercury, and 11 trace elements in Tables 4 to 8. Reference values are noncertified values that are the best estimate of the true value. However, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods.

**Expiration of Certification:** The certification of this SRM lot is valid until **01 March 2013**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is invalid if the SRM is damaged, contaminated, or modified.

**Maintenance of SRM Certification:** NIST will monitor this SRM over the period of its certification. If substantive changes occur which affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

The coordination of the technical measurements leading to the certification of this material was under the leadership of M.M. Schantz and S.A. Wise of the NIST Analytical Chemistry Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by J.C. Colbert and B.S. MacDonald of the NIST Measurement Services Division.

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Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh of the NIST Statistical Engineering Division.

Collection and preparation of SRM 1974b were performed by M.P. Cronise and C.N. Fales of the NIST Standard Reference Materials Program and P.R. Becker, E.A. Mackey, B.J. Porter, R.S. Pugh, and W.D.J. Struntz of the NIST Analytical Chemistry Division. The mussels were collected with the assistance of W. Truly of Battelle Ocean Sciences Laboratory in Duxbury, MA.

Analytical measurements for the certification of SRM 1974b were performed at NIST by J.R. Kucklick, S.E. Long, B.J. Porter, D.L. Poster, and M.M. Schantz of the NIST Analytical Chemistry Division. Results were also used from laboratories that participated in the 2000 NIST Intercomparison Exercise for Organic Contaminants in the Marine Environment [3] coordinated by M.M. Schantz and from selected laboratories that participated in the 14th Intercomparison for Trace Elements in Marine Sediments and Biological Tissues [4] coordinated by S. Willie of the National Research Council (NRC) of Canada (see Appendix A for participating laboratories). Measurements for selected trace elements were performed at NRC Canada by J.W.H. Lam, C. Scriver, S. Willie, and L. Yang. Measurements for total mercury and methylmercury were performed at the Jožef Stefan Institute (Ljubljana, Slovenia) by M. Horvat, D. Gibiĉar, and Z. Kljakovic.

## NOTICE AND WARNING TO USERS

**Storage:** SRM 1974b is packaged as a frozen tissue homogenate in glass bottles. The tissue homogenate should not be allowed to thaw prior to subsampling for analysis. If the tissue homogenate does thaw, the entire bottle should be used for analysis. This material has been stored at NIST at -80 °C (or lower) since it was prepared and should be stored by the user at this temperature, if possible, since the validity of the certified values is unknown when stored at higher temperatures.

**Handling:** This material is a frozen tissue homogenate. After extended storage at temperatures of -25 °C or higher, or if allowed to warm, the tissue homogenate will lose its powder-like form. For the handling of this material during sample preparation, the following procedures and precautions are recommended. If weighing relatively large quantities, remove a portion from the bottle and reweigh the bottle to determine the weight of the subsample. (Avoid heavy frost buildup by handling the bottles rapidly and wiping them prior to weighing.) For weighing, transfer subsamples to a pre-cooled thick-walled glass container rather than a thin-walled plastic container to minimize heat transfer to the sample. If possible, use a cold work space, e.g., an insulated container with dry ice or liquid nitrogen coolant on the bottom and pre-cooled implements, such as Teflon© coated spatulas, for transferring the powder. Normal biohazard safety precautions for the handling of biological tissues should be exercised.

**Instructions for Use:** Subsamples of this SRM for analysis should be withdrawn from the bottle immediately after opening and used without delay for the certified values listed in Tables 1 to 3 to be valid within the stated uncertainties. The concentrations of constituents in SRM 1974b are reported on both a wet-mass and a dry-mass basis for user convenience. The SRM tissue homogenate, as received, contains approximately 90 % moisture. A separate subsample of the SRM should be removed from the bottle at the time of analysis and dried to determine the concentration on a dry-mass basis.

## PREPARATION AND ANALYSIS[1]

**Sample Collection and Preparation:** The mussels (*Mytilus edulis*) used for the preparation SRM 1974b were collected October 27, 1999 from Dorchester Bay within Boston (MA) Harbor (42°18.25'N and 72°02.31'W) following the same procedures as described previously for the collection of mussels for SRM 1974 and SRM 1974a [5,6]. Approximately 6300 individual mussels were collected by hand at low tide. The samples were transported to the Battelle Ocean Sciences Laboratory (Duxbury, MA) where the mussels were rinsed with water to remove rocks and other debris. The samples were placed in insulted Teflon©-lined wooden containers, frozen, and transported to NIST on dry ice. The samples were transferred to Teflon© bags and stored in a liquid nitrogen vapor freezer (-120 °C) until they were shucked.

[1] Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

**Sample Preparation:** The mussel tissue was removed from the shell using the following procedure. The mussels were allowed to warm up to about 0 °C; the tissue was removed from the shell using a titanium knife and placed in Teflon© bags (approximately 0.5 kg per bag) and immediately returned to a liquid nitrogen freezer. Approximately 59 kg of mussel tissue was prepared for use as the SRM. The frozen mussel tissue was pulverized in batches of approximately 700 g each using a cryogenic procedure described previously [7]. The pulverized material was then homogenized in an aluminum mixing drum in two batches of approximately 30 kg each. The mixing drum was designed to fit inside the liquid nitrogen vapor freezer and to rotate in the freezer thereby mixing the frozen tissue powder. After mixing for 2 h, subsamples (approximately 8 g to 10 g) of the mussel tissue homogenate were aliquoted into cleaned, pre-cooled glass bottles.

**Conversion to Dry-Mass Basis:** The moisture content of the mussel homogenate was determined by measuring the mass loss after freeze drying. Ten bottles of SRM 1974b were selected according to a stratified randomization scheme for the drying study. The entire contents of each glass bottle were transferred to a Teflon© bottle and dried for seven days at 1 Pa with a -20 °C shelf temperature and a -50 °C condenser temperature. The moisture content in SRM 1974b at the time of the certification analyses was 89.87 % ± 0.05 % (95 % confidence level). Analytical results for the organic constituents were determined on a wet-mass basis and then converted to a dry-mass basis by dividing by the conversion factor of 0.1013 (g dry mass/g wet mass). The trace elements, other than mercury, were determined on a dry-mass basis and then converted to a wet-mass basis by multiplying by the conversion factor of 0.1013 (g dry mass/g wet mass).

**Polycyclic Aromatic Hydrocarbons:** The general approach used for the value assignment of the PAHs in SRM 1974b was similar to that reported for the recent certification of several environmental matrix SRMs [6,8,9,10] and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and pressurized fluid extraction (PFE) using dichloromethane (DCM) or a hexane/acetone mixture, cleanup of the extracts using size exclusion chromatography (SEC) and/or solid phase extraction (SPE), followed by analysis using gas chromatography/mass spectrometry (GC/MS) analysis of the PAH fraction on two stationary phases of different selectivity, i.e., a 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase and a relatively non-polar proprietary phase.

Six sets of GC/MS results, designated as GC/MS (I) through GC/MS (V) were obtained using two columns with different selectivities for the separation of PAHs. For GC/MS (I) analyses, duplicate subsamples of between 2 g and 3 g from 10 bottles of SRM 1974b were extracted using PFE with 50 % hexane and 50 % acetone (volume fraction) [11]. The concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. Following concentration, the silica SPE step was repeated. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a relatively non-polar proprietary phase (0.25 µm film thickness) (DB-XLB, J&W Scientific, Folsom, CA). This method is designated as GC/MS (Ia). For GC/MS (1b), the same extracts were analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-17MS, J&W Scientific, Folsom, CA). The GC/MS (II) analyses were performed using subsamples of 8 g to 10 g from six bottles of SRM 1974b. These samples were extracted using PFE with DCM. The high molecular mass compounds (i.e, lipids and biogenic material) were removed from the extracts using SEC with a preparative-scale divinylbenzene-polystyrene column (10 µm particle size with 100 Å diameter pores), and the concentrated extract was passed through an aminopropyl SPE cartridge and eluted with 10 % DCM in hexane. GC/MS analysis was performed using a 0.25 mm i.d. × 60 m fused silica capillary column with a 50 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-17MS). For the GC/MS (III) analyses, approximately 10 g subsamples from six bottles of SRM 1974b were Soxhlet extracted for 18 h with 250 mL of DCM. The extracts was cleaned up using SEC as described above, and the concentrated extract was passed through a silica SPE cartridge and eluted with 2 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a relatively non-polar proprietary phase (0.25 µm film thickness) (DB-XLB) and a 50 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-17 MS). The GC/MS (IV) method used 9 g subsamples from three bottles of SRM 1974b with the same clean-up and analysis method as GC/MS (Ia) while the GC/MS (V) method used 9 g subsamples from three bottles of SRM 1974b with the same clean-up and analysis method as GC/MS (II). For the GC/MS measurements described above, selected perdeuterated PAHs were added to the mussel tissue homogenate prior to solvent extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 1974b was used in an interlaboratory comparison exercise in 2000 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [3]. Results from 16 laboratories that participated in this exercise were used as the seventh data set in the determination of the

certified values for PAHs in SRM 1974b. The laboratories participating in this exercise employed the analytical procedures routinely used in their laboratories to measure PAHs.

**Homogeneity Assessment for PAHs:** The homogeneity of SRM 1974b was assessed by analyzing duplicate samples of between 2 g and 3 g from 10 bottles selected by stratified random sampling. Samples were extracted, processed, and analyzed as described above for GC/MS (Ia and Ib). No statistically significant differences among bottles were observed for the PAHs at this sample size.

**PCBs and Chlorinated Pesticides:** The general approach used for the determination of PCBs and chlorinated pesticides in SRM 1974b was similar to that reported for the recent certification of several environmental matrix SRMs [6,8-10,12-14], and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and PFE using DCM or a hexane/acetone mixture, cleanup/isolation using SEC, SPE or liquid chromatography (LC), followed by analysis using GC/MS and gas chromatography with electron capture detection (GC-ECD) on three columns with different selectivity for the separation of PCBs and chlorinated pesticides.

Eight sets of results were obtained designated as GC/MS (Ia and Ib), GC/MS (II), GC-ECD (Ia and Ib), GC-ECD (II), GC-ECD (III), and Interlaboratory Comparison Exercise. For GC/MS (Ia and Ib), duplicate subsamples of between 2 g and 3 g from 10 bottles of SRM 1974b were extracted using PFE with 50 % hexane and 50 % acetone (volume fraction). The concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. Following concentration of the extract, the silica SPE step was repeated. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a relatively non-polar proprietary phase (0.25 µm film thickness) (DB-XLB). This method is designated as GC/MS (Ia). For GC/MS (1b), the same extracts were analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-17MS). For GC/MS (II), subsamples of 9 g from three bottles of SRM 1974b were extracted using Soxhlet extraction with DCM. The concentrated extracts were processed as described above for GC/MS I and then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a relatively nonpolar proprietary phase (0.25 µm film thickness) (DB-XLB, J&W Scientific, Folsom, CA). For the GC/MS analyses, selected carbon-13 labeled PCB congeners and chlorinated pesticides were added to the mussel tissue homogenate prior to extraction for use as internal standards for quantification purposes.

For GC-ECD (Ia and Ib), subsamples of between 8 g and 10 g from six bottles of SRM 1974b were extracted using PFE with DCM, followed by SEC, as described above for the PAHs, to remove the high molecular mass compounds. The concentrated extracts were then passed through an aminopropyl SPE cartridge and eluted with 10 % DCM in hexane. The concentrated extract was fractionated on a semi-preparative aminopropylsilane LC column to isolate two fractions containing: (1) the PCBs and lower polarity pesticides and, (2) the more polar pesticides. GC-ECD analyses of the two fractions were performed on two columns of different selectivities for PCB separations: 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5, J&W Scientific, Folsom, CA) and a 0.25 mm × 60 m fused silica capillary column with a nonpolar proprietary phase (0.25 µm film thickness) (DB-XLB). The results from the 5 % phenyl phase are designated as GC-ECD (Ia) and the results from the proprietary phase are designated as GC-ECD (Ib). The GC-ECD (II) analyses used Soxhlet extraction with DCM followed by SEC to remove the high molecular mass compounds and fractionation of the extract using the semi-preparative aminopropylsilane LC column described for GC-ECD (I). The GC-ECD analysis used a 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5). The GC-ECD (III) method used 9 g subsamples from three bottles of SRM 1974b extracted, processed, and analyzed as described above for GC-ECD (I). For the GC-ECD analyses, two PCB congeners that are not significantly present in the mussel tissue extract (PCB 103 and PCB 198 [25,26]), and endosulfan I-$d_4$, 4,4'-DDE-$d_8$, 4,4'-DDD-$d_8$, and 4,4'-DDT-$d_8$ were added to the mussel tissue homogenate prior to extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 1974b was used in an interlaboratory comparison exercise in 2000 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [3]. Results from 16 laboratories that participated in this exercise were used as the eighth data set in the determination of the certified values for PCB congeners and chlorinated pesticides in SRM 1974b. The laboratories participating in this exercise employed the analytical procedures routinely used in their laboratories to measure PCB congeners and chlorinated pesticides.

The reference value for PCB 77 (3,3',4,4'-tetrachlorobiphenyl) was determined from the GC-ECD (I) samples. The first fraction (PCBs and lower polarity pesticides) from the semi-preparative aminopropylsilane column was further fractionated using a Cosmosil PYE column (5 µm particle size, 4.6 mm i.d. × 25 cm, Phenomenex, Torrance, CA) [15].

Three fractions were collected: the first fraction contained the pesticides and multi-*ortho* PCBs, the second fraction contained the polychlorinated naphthalenes, non-*ortho* PCB congeners, and some mono-*ortho* PCB congeners, and the third fraction removed the residual planar compounds from the column. The second fraction was analyzed by GC/MS using a 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5MS, J&W Scientific, Folsom, CA). Carbon-13 labeled PCB 77 was used as an internal standard for quantification purposes.

**Homogeneity Assessment for PCBs and Chlorinated Pesticides:** The homogeneity of SRM 1974b was assessed by analyzing duplicate samples of between 2 g and 3 g from 10 bottles selected by stratified random sampling. Samples were extracted, processed, and analyzed as described above for GC/MS (Ia and Ib). No statistically significant differences among bottles were observed for the chlorinated analytes at this sample size.

**Total PCBs and Total Extractable Organics:** A subset of laboratories participated in an interlaboratory comparison study for total PCBs and total extractable organics (TEO) in SRM 1974b. The methods used by the four laboratories reporting total PCBs were: sum of congeners using GC/MS; determination of 112 congeners using GC-ECD; calibration of GC-ECD using Aroclors 1242, 1248, 1254, and 1260; and use of an individual congener for each homolog group to calibrate the GC/MS and then summing the homolog groups.

The TEO values were determined gravimetrically by six laboratories after extraction using the following conditions: PFE with DCM (2 laboratories), Soxhlet extraction with DCM (2 laboratories), Soxhlet extraction with hexane (1 laboratory), and PFE with a DCM/acetone mixture (1 laboratory).

**Methylmercury and Total Mercury:** The certified value for total mercury is based on results of analyses of SRM 1974b at NIST, the Jožef Stefan Institute (Ljubljana, Slovenia), NRC Canada, and selected participants in an interlaboratory comparison exercise coordinated by NRC Canada. For total mercury measurements at NIST, subsamples of »500 mg from six bottles of SRM 1974b were analyzed. The analytical procedure consisted of spiking with $^{201}Hg$ as an internal standard, microwave-assisted acid digestion of the tissue, followed by cold vapor generation coupled with inductively coupled plasma mass spectrometry (CV-ICP-MS) isotope ratio measurements as described previously [16]. At the Jožef Stefan Institute triplicate subsamples (»500 mg) from six bottles of SRM 1974b were digested with acid and analyzed by cold vapor atomic absorption spectrometry (CVAAS) [17,18]. At NRC Canada, total mercury was determined by analyzing five subsamples (»250 mg dry mass) using microwave-assisted acid digestion followed by CVAAS. Results from four selected laboratories participating in the NRC Canada intercomparison exercise [4] (see below) were also used in the value assignment for total mercury.

The reference value for methylmercury is based on results from two methods performed at the Jožef Stefan Institute. For the first method, triplicate subsamples (»500 mg) from six bottles of SRM 1974b were analyzed using solid-liquid extraction into toluene followed by GC-ECD [19,20]. The second analytical method for methylmercury (subsamples of »500 mg from six bottles) consisted of acid digestion, anion exchange chromatographic separation of inorganic mercury and methylmercury, followed by CVAAS detection before and after ultraviolet radiation [21,22].

**Additional Trace Element Analyses:** SRM 1974b was freeze-dried and used in an interlaboratory comparison study coordinated by the NRC Canada [4]. The laboratories participating in this exercise employed the analytical procedures routinely used in their laboratories to measure the selected trace elements. Value assignment for the concentrations of the trace elements was accomplished by combining the results from the analyses of the freeze-dried sample of SRM 1974b from (1) NRC Canada using isotope dilution ICP-MS, graphite furnace atomic absorption spectrometry (GFAAS), and/or inductively coupled plasma atomic emission spectroscopy (ICP-AES) and (2) the mean of the results from six selected laboratories that participated in the NRC Canada interlaboratory study [4] using a variety of analytical techniques (laboratories listed in Appendix A).

Table 1. Certified Concentrations for Selected PAHs in SRM 1974b

| | Mass Fractions in µg/kg[a] | | | | | |
|---|---|---|---|---|---|---|
| | Wet-Mass Basis | | | Dry-Mass Basis | | |
| Naphthalene[d,e,f,g,h,i,j] | 2.43 | ± | 0.12[b] | 24.0 | ± | 1.2[b] |
| Fluorene[d,e,f,g,h,i,j] | 0.494 | ± | 0.036[b] | 4.88 | ± | 0.36[b] |
| Phenanthrene[d,e,f,g,h,i ,j] | 2.58 | ± | 0.11[b] | 25.5 | ± | 1.1[b] |
| Anthracene[d,e,f,g,h,i,j] | 0.527 | ± | 0.071[c] | 5.20 | ± | 0.71[c] |
| 1-Methylphenanthrene[d,e,f,g,h,i,j] | 0.98 | ± | 0.13[c] | 9.66 | ± | 1.3[c] |
| 2-Methylphenathrene[d,e,f,g] | 1.28 | ± | 0.31[b] | 24.0 | ± | 1.2[b] |
| 3-Methylphenanthrene[d,e,g] | 1.27 | ± | 0.04[c] | 12.5 | ± | 0.4[c] |
| Fluoranthene[d,e,f,g,h,i,j] | 17.1 | ± | 0.7[b] | 169 | ± | 7[b] |
| Pyrene[d,e,f,g,h,i,j] | 18.04 | ± | 0.6[b] | 178 | ± | 6[b] |
| Benz[*a*]anthracene[d,e,f,g,h,i,j] | 4.74 | ± | 0.53[b] | 46.8 | ± | 5.2[b] |
| Chrysene[d,g,h] | 6.3 | ± | 1.0[b] | 62.2 | ± | 9.9[b] |
| Triphenylene[d,g,h] | 4.33 | ± | 0.72[b] | 42.7 | ± | 7.1[b] |
| Benzo[*b*]fluoranthene[e,f,g,h,i,j] | 6.46 | ± | 0.59[b] | 63.8 | ± | 5.8[b] |
| Benzo[*j*]fluoranthene[e,f,g,h,i] | 2.99 | ± | 0.29[b] | 29.5 | ± | 2.9[b] |
| Benzo[*k*]fluoranthene[d,e,f,g,h,i,j] | 3.16 | ± | 0.18[b] | 31.2 | ± | 1.8[b] |
| Benzo[*a*]fluoranthene[d,e,f,g] | 0.634 | ± | 0.074[b] | 6.26 | ± | 0.73[b] |
| Benzo[*e*]pyrene[d,e,f,g,h,i,j] | 10.3 | ± | 1.1[b] | 102 | ± | 11[b] |
| Benzo[*a*]pyrene[d,e,f,g,h,i,j] | 2.80 | ± | 0.38[b] | 27.6 | ± | 3.8[b] |
| Perylene[d,e,f,g,h,i,j] | 0.99 | ± | 0.14[b] | 9.8 | ± | 1.4[b] |
| Benzo[*ghi*]perylene[d,e,f,g,h,i,j] | 3.12 | ± | 0.33[b] | 30.8 | ± | 3.3[b] |
| Indeno[1,2,3-*cd*]pyrene[d,e,f,g,h,i,j] | 2.14 | ± | 0.11[b] | 21.1 | ± | 1.1[b] |
| Dibenz[*a,h*]anthracene[e,f,g,h,i] | 0.327 | ± | 0.031[c] | 3.23 | ± | 0.31[c] |

[a] Concentrations reported on both wet- and dry-mass basis; material as received contains 89.87 % ± 0.05 % (95 % confidence level) water.

[b] Certified values are weighted means of the results from three to seven analytical methods [23]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-source variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurements [2].

[c] The certified value is an unweighted mean of the results from three to seven analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [24] with a pooled, within method variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurement [2]. Note for anthracene and 1-methylphenanthrene the within method variance for the interlaboratory study was not used for the calculation of the expanded uncertainty.

[d] GC/MS (Ia) on a relatively nonpolar proprietary phase after PFE with 50 % hexane/50 % acetone mixture.

[e] GC/MS (Ib) on 50 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (Ia).

[f] GC/MS (II) on 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[g] GC/MS (III) on a relatively nonpolar proprietary phase and 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

[h] GC/MS (IV) on a relatively nonpolar proprietary phase after Soxhlet extraction with DCM.

[i] GC/MS (V) on 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[j] 2000 NIST Intercomparison Exercise for Organic Contaminants in the Marine Environment [3] with 16 laboratories submitting data.

Table 2. Certified Concentrations for Selected PCB Congeners[a] in SRM 1974b

| | | Mass Fractions in µg/kg[b] | |
|---|---|---|---|
| | | Wet-Mass Basis | Dry-Mass Basis |
| PCB 18 | (2,2',5-Trichlorobiphenyl)[e,f,g,h,i,j,k,l] | 0.84 ± 0.13[c] | 8.30 ± 1.3[c] |
| PCB 28 | (2,4,4'-Trichlorobiphenyl)[e,f,g,h,j.k.l] | 3.43 ± 0.25[c] | 33.9 ± 2.5[c] |
| PCB 31 | (2,4',5-Trichlorobiphenyl)[e,f,g,h,i,j,k,l] | 2.88 ± 0.23[c] | 28.4 ± 2.3[c] |
| PCB 44 | (2,2'3,5'-Tetrachlorobiphenyl)[e,f,g,h,i,j,k,l] | 3.85 ± 0.20[c] | 38.0 ± 2.0[c] |
| PCB 49 | (2,2'4,5'-Tetrachlorobiphenyl)[e,f,g,h,i,j,k,l] | 5.66 ± 0.23[c] | 55.9 ± 2.3[c] |
| PCB 52 | (2,2',5,5'-Tetrachlorobiphenyl)[e,f,g,h,i,j,k,l] | 6.26 ± 0.37[c] | 61.8 ± 3.7[c] |
| PCB 66 | (2,3',4,4'-Tetrachlorobiphenyl)[e,f,g,h,j,k,l] | 6.37 ± 0.37[c] | 62.9 ± 3.7[c] |
| PCB 70 | (2,3',4',5-Tetrachlorobiphenyl)[e,f,h,i] | 6.01 ± 0.22[d] | 59.3 ± 2.2[d] |
| PCB 74 | (2,4,4',5-Tetrachlorobiphenyl)[e,f,h,i] | 3.55 ± 0.23[c] | 35.0 ± 2.3[c] |
| PCB 82 | (2,2',3,3',4-Pentachlorobiphenyl)[e,f,g,i] | 1.16 ± 0.14[c] | 11.5 ± 1.4[c] |
| PCB 87 | (2,2',3,4,5'-Pentachlorobiphenyl)[e,f,i] | 4.33 ± 0.36[d] | 42.7 ± 3.6[d] |
| PCB 95 | (2,2',3,5',6-Pentachlorobiphenyl)[e,f,g,h,j,k,l] | 6.04 ± 0.36[c] | 59.6 ± 3.6[c] |
| PCB 99 | (2,2',4,4',5-Pentachlorobiphenyl)[e,f,g,h,i,j,k,l] | 5.92 ± 0.27[c] | 58.4 ± 2.7[c] |
| PCB 101 | (2,2',4,5,5'-Pentachlorobiphenyl)[e,f,h,i,j,k,l] | 10.7 ± 1.1[c] | 106 ± 11[c] |
| PCB 105 | (2,3,3',4,4'-Pentachlorobiphenyl)[e,f,g,h,i,j,k,l] | 4.00 ± 0.18[c] | 39.5 ± 1.8[c] |
| PCB 107 | (2,3,3',4,5'-Pentachlorobiphenyl)[e,f,g,h,i] | 1.03 ± 0.12[c] | 10.2 ± 1.2[c] |
| PCB 110 | (2,3,3',4',6-Pentachlorobiphenyl)[e,f,h] | 10.0 ± 0.7[c] | 99.1 ± 7.1[c] |
| PCB 118 | (2,3',4,4',5-Pentachlorobiphenyl)[e,f,g,h,i,j,k,l] | 10.3 ± 0.4[c] | 102 ± 4[c] |
| PCB 128 | (2,2',3,3',4,4'-Hexachlorobiphenyl)[e,f,g,h,i,j,k,l] | 1.79 ± 0.12[c] | 17.7 ± 1.2[c] |
| PCB 132 | (2,2',3,3',4,6'-Hexachlorobiphenyl)[e,f,g,h,i] | 2.43 ± 0.25[c] | 24.0 ± 2.5[c] |
| PCB 138 | (2,2',3,4,4',5'-Hexachlorobiphenyl)[e,f,h,j,k,l] | 9.2 ± 1.4[c] | 91 ± 14[c] |
| PCB 146 | (2,2',3,4',5,5'-Hexachlorobiphenyl)[e,f,g,h] | 1.92 ± 0.16[c] | 19.0 ± 1.6[c] |
| PCB 149 | (2,2',3,4',5',6-Hexachlorobiphenyl)[e,f,h,i,j,k,l] | 7.01 ± 0.28[c] | 69.2 ± 2.8[c] |
| PCB 151 | (2,2',3,5,5',6-Hexachlorobiphenyl)[e,f,g,i] | 1.86 ± 0.16[c] | 18.4 ± 1.6[c] |
| PCB 153 | (2,2',4,4',5,5'-Hexachlorobiphenyl)[e,f,g,h,i,j,k,l] | 12.3 ± 0.8[c] | 121 ± 8[c] |
| PCB 156 | (2,3,3',4,4',5-Hexachlorobiphenyl)[e,f,h,j,k,l] | 0.718 ± 0.080[c] | 7.09 ± 0.79[c] |
| PCB 158 | (2,3,3',4,4',6-Hexachlorobiphenyl)[e,g,h,i] | 0.999 ± 0.096[c] | 9.86 ± 0.95[c] |
| PCB 170 | (2,2',3,3',4,4',5-Heptachlorobiphenyl)[e,f,h,j,k,l] | 0.269 ± 0.034[c] | 2.66 ± 0.34[c] |
| PCB 180 | (2,2',3,4,4',5,5'-Heptachlorobiphenyl)[e,f,g,h,i,j,k,l] | 1.17 ± 0.10[c] | 11.5 ± 1.0[c] |
| PCB 183 | (2,2',3,4,4',5',6-Heptachlorobiphenyl)[e,f,g,h,i] | 1.25 ± 0.03[c] | 12.3 ± 0.3[c] |
| PCB 187 | (2,2',3,4',5,5',6-Heptachlorobiphenyl)[e,f,g,h,i,j,k,l] | 2.94 ± 0.15[c] | 29.0 ± 1.5[c] |

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [25] and later revised by Schulte and Malisch [26] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, only PCB 107 is different in the numbering systems. Under the Ballschmiter and Zell numbering system, the IUPAC PCB 107 is listed as PCB 108.

[b] Concentrations reported on both wet- and dry-mass basis; material as received contains 89.87 % ± 0.05 % (95 % confidence level) water.

[c] Certified values are weighted means of the results from three to eight analytical methods [23]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-source variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurements [2].

[d] The certified value is an unweighted mean of the results from three analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [24] with a pooled, within method variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurement [2].

[e] GC/MS (Ia) on a relatively nonpolar proprietary phase after PFE with 50 % hexane/50 % acetone mixture.

[f] GC/MS (Ib) on 50 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (Ia).

[g] GC-ECD (Ia) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[h] GC-ECD (Ib) on a relatively nonpolar proprietary phase; same extracts as GC-ECD (Ia).

[i] GC-ECD (II) on a 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

[j] GC/MS (II) on a relatively nonpolar proprietary phase after Soxhlet extraction with DCM.

[k] GC-ECD (III) on a 5 % phenyl-substituted methylpolysiloxane phase and a relatively non-polar proprietary phase after PFE with DCM.

[l] 2000 NIST Intercomparison Exercise for Organic Contaminants in the Marine Environment [3] with 16 laboratories submitting data.

Table 3. Certified Concentrations for Selected Chlorinated Pesticides in SRM 1974b

| | Mass Fractions in µg/kg[a,b] | | | | | |
|---|---|---|---|---|---|---|
| | Wet-Mass Basis | | | Dry-Mass Basis | | |
| *cis*-Chlordane[c,d,e,f,g,h,i,j] | 1.36 | ± | 0.10 | 13.4 | ± | 1.0 |
| *trans*-Chlordane[c,d,e,f,g,h,i,j] | 1.14 | ± | 0.17 | 11.3 | ± | 1.7 |
| *trans*-Nonachlor[c,d,e,f,g,h,i,j] | 1.30 | ± | 0.14 | 12.8 | ± | 1.4 |
| 2,4'-DDE[c,d,h,i,j] | 0.336 | ± | 0.044 | 3.32 | ± | 0.43 |
| 4,4'-DDE[c,d,e,f,g,h,i,j] | 4.15 | ± | 0.38 | 41.0 | ± | 3.8 |
| 2,4'-DDD[c,d,e,f,h,i,j] | 1.09 | ± | 0.16 | 10.8 | ± | 1.6 |
| 4,4'-DDD[c,d,e,f,g,h,i,j] | 3.34 | ± | 0.22 | 33.0 | ± | 2.2 |

[a] Concentrations reported on both wet- and dry-mass basis; material as received contains 89.87 % ± 0.05 % (95 % confidence level) water.

[b] Certified values are weighted means of the results from five to eight analytical methods [23]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-source variance incorporating inter-method bias with a pooled within-source variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurements [2].

[c] GC/MS (Ia) on a relatively non-polar proprietary phase after PFE with 50 % hexane/50 % acetone mixture.

[d] GC/MS (Ib) on 50 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (Ia).

[e] GC-ECD (Ia) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[f] GC-ECD (Ib) on a relatively non-polar proprietary phase; same extracts as GC-ECD (Ia).

[g] GC-ECD (II) on a 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

[h] GC/MS (II) on a relatively non-polar proprietary phase after Soxhlet extraction with DCM.

[i] GC-ECD (III) on a 5 % phenyl-substituted methylpolysiloxane phase and a relatively non-polar proprietary phase after PFE with DCM.

[j] 2000 NIST Intercomparison Exercise for Organic Contaminants in the Marine Environment [3] with 16 laboratories submitting data.

Table 4. Certified and Reference Concentrations for Total Mercury and Methylmercury in SRM 1974b

| | Mass Fraction in µg/kg[a] | | | | | |
|---|---|---|---|---|---|---|
| | Wet-Mass Basis | | | Dry-Mass Basis | | |
| Total Mercury[b] | 17.0 | ± | 1.1[b] | 167 | ± | 11[b] |
| Methylmercury[c] | 7.05 | ± | 0.44[c] | 69.6 | ± | 4.3[c] |

[a] The concentrations are reported on both wet- and dry-mass basis; material as received contains 89.87 % ± 0.05 % (95 % confidence level) water.

[b] The certified value for total mercury is the weighted mean of four results [23] from the following: (1) ICP-MS analyses performed at NIST, (2) ICP-MS analyses performed at NRC Canada, (3) the mean of results from four selected laboratories participating in the NRC Canada 14th Intercomparison for Trace Elements in Marine Sediments and Biological Tissues [4], and (4) results from CV-AAS performed at the Jožef Stefan Institute. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-source variance incorporating inter-method bias with a pooled within-source variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurements [2].

[c] The reference value for methylmercury is an unweighted mean of the results from CV-AAS and GC-ECD performed at the Jožef Stefan Institute. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [24] with a pooled, within method variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurement [2].

Table 5. Reference Concentrations for Selected PAHs in SRM 1974b

| | Mass Fractions in µg/kg[a] | |
|---|---|---|
| | Wet-Mass Basis | Dry-Mass Basis |
| 1-Methylnaphthalene[e,f,g,h,i,j,k] | 0.614 ± 0.050[b] | 6.06 ± 0.49[b] |
| 2-Methylnaphthalene[e,f,g,h,i,j,k] | 1.25 ± 0.09[b] | 12.3 ± 0.9[b] |
| 2,6-Dimethylnaphthalene[e,f,g,h,i,j,k] | 0.33 ± 0.16[b] | 3.3 ± 1.6[b] |
| 2,3,5-Trimethylnaphthalene[e,f,g,h,i,j,k] | 0.400 ± 0.032[b] | 3.95 ± 0.32[b] |
| Biphenyl[e,f,g,h,i,j,k] | 0.61 ± 0.14[b] | 6.0 ± 1.4[b] |
| Acenaphthylene[e,f,g,h,i,j,k] | 0.48 ± 0.12[b] | 4.7 ± 1.2[b] |
| Acenaphthene[e,f,g,h,i,j,k] | 0.274 ± 0.054[b] | 2.70 ± 0.53[b] |
| 4-Methylphenanthrene and 9-Methylphenanthrene[g,h] | 1.60 ± 0.18[b] | 15.8 ± 1.8[b] |
| 2-Methylanthracene[e,f] | 0.232 ± 0.004[c] | 2.29 ± 0.04[c] |
| Cyclopenta[*cd*]pyrene[h] | 0.227 ± 0.010[d] | 2.24 ± 0.10[d] |
| Benzo[*c*]phenanthrene[e,f,h] | 1.85 ± 0.21[b] | 18.3 ± 2.1[b] |
| Benzo[*b*]chrysene[h] | 0.507 ± 0.030[d] | 5.00 ± 0.30[d] |
| Benzo[*c*]chrysene[g,h] | 0.318 ± 0.042[b] | 3.14 ± 0.42[b] |
| Dibenz[*a,c*]anthracene[f,g] | 0.212 ± 0.013[c] | 2.09 ± 0.13[c] |
| Dibenz[*a,j*]anthracene[g,h] | 0.467 ± 0.048[b] | 4.61 ± 0.47[b] |
| Picene[g,h] | 0.75 ± 0.16[b] | 7.4 ± 1.6[b] |

[a] Concentrations reported on both wet- and dry-mass basis; material as received contains 89.87 % ± 0.05 % (95 % confidence level) water.

[b] The reference value is a weighted mean of the results from two to seven analytical methods [23]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-source variance incorporating inter-method bias with a pooled within-source variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurements [2].

[c] The reference value is an unweighted mean of the results from two analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [24] with a pooled, within method variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurement [2].

[d] The reference value is the mean of results obtained by NIST using one analytical technique. The expanded uncertainty, $U$, is calculated as $U = ku_c$, where $u_c$ is intended to represent, at the level of one standard deviation, the combined standard uncertainty calculated according to the ISO Guide [2]. The coverage factor, $k$, is determined from the Student's $t$-distribution corresponding to the appropriate associated degrees of freedom and 95 % confidence for each analyte.

[e] GC/MS (Ia) on a relatively nonpolar proprietary phase after PFE with 50 % hexane/50 % acetone mixture.

[f] GC/MS (Ib) on 50 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (Ia).

[g] GC/MS (II) on 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[h] GC/MS (III) on a relatively nonpolar proprietary phase and 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

[i] GC/MS (IV) on a relatively nonpolar proprietary phase after Soxhlet extraction with DCM.

[j] GC/MS (V) on 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[k] 2000 NIST Intercomparison Exercise for Organic Contaminants in the Marine Environment [3] with 16 laboratories submitting data.

Table 6. Reference Concentrations for Selected PCB Congeners[a] and Total PCBs in SRM 1974b

| | | Mass Fractions in µg/kg [b] | |
|---|---|---|---|
| | | Wet-Mass Basis | Dry-Mass Basis |
| PCB 8 | (2,4'-Dichlorobiphenyl)[f,g] | 0.37 ± 0.11[c] | 3.7 ± 1.1[c] |
| PCB 45 | (2,2',3,6-Tetrachlorobiphenyl)[f,h,i,j] | 0.50 ± 0.18[d] | 4.9 ± 1.8[d] |
| PCB 56 | (2,3,3',4-Tetrachlorobiphenyl)[f,h,i,k] | 2.82 ± 0.56[d] | 27.8 ± 5.5[d] |
| PCB 63 | (2,3,4',5-Tetrachlorobiphenyl)[f,h,j,k] | 0.46 ± 0.14[d] | 4.5 ± 1.4[d] |
| PCB 77 | (3,3',4,4'-Tetrachlorobiphenyl)[l] | 0.563 ± 0.023[e] | 5.56 ± 0.23[e] |
| PCB 92 | (2,2',3,5,5'-Pentachlorobiphenyl)[f,h,i,k] | 2.76 ± 0.58[d] | 27.2 ± 5.7[d] |
| PCB 157 | (2,3,3',4,4',5'-Hexachlorobiphenyl)[f,h,i] | 0.236 ± 0.024[d] | 2.33 ± 0.24[d] |
| PCB 163 | (2,3,3',4',5,6-Hexachlorobiphenyl)[f,h,i] | 2.02 ± 0.05[c] | 19.9 ± 0.5[c] |
| Total PCBs[m] | | 205 ± 42 | 2020 ± 420 |

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [25] and later revised by Schulte and Malisch [26] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, only PCB 107 (Table 2) is different in the numbering systems. Under the Ballschmiter and Zell numbering system, the IUPAC PCB 107 is listed as PCB 108.

[b] Concentrations reported on both wet- and dry-mass basis; material as received contains 89.87 % ± 0.05 % (95 % confidence level) water.

[c] The reference value is an unweighted mean of the results from two to three analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [24] with a pooled, within method variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurement [2].

[d] The reference value is a weighted mean of the results from three to four analytical methods [23]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-source variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurements [2].

[e] The reference value is the mean of results obtained by NIST using one analytical technique. The expanded uncertainty, $U$, is calculated as $U = ku_c$, where $u_c$ is intended to represent, at the level of one standard deviation, the combined standard uncertainty calculated according to the ISO Guide [2]. The coverage factor, $k$, is determined from the Student's $t$-distribution corresponding to the appropriate associated degrees of freedom and 95 % confidence for the analyte.

[f] GC-ECD (Ib) on a relatively nonpolar proprietary phase; same extracts as GC-ECD (Ia).

[g] 2000 NIST Intercomparison Exercise for Organic Contaminants in the Marine Environment [3] with 16 laboratories submitting data.

[h] GC/MS (Ia) on a relatively nonpolar proprietary phase after PFE with 50 % hexane/50 % acetone mixture.

[i] GC/MS (Ib) on 50 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (Ia).

[j] GC-ECD (Ia) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[k] GC-ECD (II) on a 5% phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

[l] GC/MS on a 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (I) fractionated using a PYE column.

[m] Interlaboratory comparison study with four laboratories submitting data (See Preparation and Analysis for definition of total PCBs.). The expanded uncertainty, $U$, is calculated as $U = ku_c$, where $u_c$ is intended to represent, at the level of one standard deviation, the combined standard uncertainty calculated according to the ISO Guide [2]. The coverage factor, $k$, is determined from the Student's $t$-distribution corresponding to the appropriate associated degrees of freedom and 95 % confidence for the total PCBs.

Table 7. Reference Concentrations for Selected Chlorinated Pesticides and Total Extractable Organics in SRM 1974b

| | Mass Fractions in µg/kg[a] | |
|---|---|---|
| | Wet-Mass Basis | Dry-Mass Basis |
| Heptachlor[d,e] | 0.212 ± 0.084[b] | 2.09 ± 0.83[b] |
| Oxychlordane[d,e] | 0.362 ± 0.072[b] | 3.57 ± 0.71[b] |
| Dieldrin[d,e,f,g,h,i] | 0.62 ± 0.13[c] | 6.1 ± 1.3[c] |
| *cis*-Nonachlor[d,e,f,g,h,i,j] | 0.64 ± 0.16[c] | 6.3 ± 1.6[c] |
| 2,4'-DDT[e,h,i] | 0.894 ± 0.057[b] | 8.83 ± 0.56[b] |
| 4,4'-DDT[d,e,f,g,h,i,j,k] | 0.396 ± 0.096[c] | 3.91 ± 0.94[c] |
| | Percent | |
| Total Extractable Organics (TEO)[l] | 0.64 ± 0.13 | 6.3 ± 1.3 |

[a] Concentrations reported on both wet- and dry-mass basis; material as received contains 89.87 % ± 0.05 % (95 % confidence level) water.

[b] The reference value is an unweighted mean of the results from two to three analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [24] with a pooled, within method variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurement [2].

[c] The reference value is a weighted mean of the results from six to eight analytical methods [23]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-source variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurements [2].

[d] GC-ECD (Ib) on a relatively nonpolar proprietary phase; same extracts as GC-ECD (Ia).

[e] GC-ECD (III) on a 5 % phenyl-substituted methylpolysiloxane phase and a relatively non-polar proprietary phase after PFE with DCM.

[f] GC/MS (Ib) on 50 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (Ia).

[g] GC-ECD (Ia) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[h] GC/MS (II) on a relatively nonpolar proprietary phase after Soxhlet extraction with DCM.

[i] 2000 NIST Intercomparison Exercise for Organic Contaminants in the Marine Environment [3] with 16 laboratories submitting data.

[j] GC/MS (Ia) on a relatively nonpolar proprietary phase after PFE with 50 % hexane/50 % acetone mixture.

[k] GC-ECD (II) on a 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

[l] Interlaboratory comparison study with six laboratories submitting data. The expanded uncertainty, $U$, is calculated as $U = ku_c$, where $u_c$ is intended to represent, at the level of one standard deviation, the combined standard uncertainty calculated according to the ISO Guide [2]. The coverage factor, $k$, is determined from the Student's $t$-distribution corresponding to the appropriate associated degrees of freedom and 95 % confidence for the TEO.

Table 8. Reference Concentrations for Additional Trace Elements in SRM 1974b

| | Mass Fraction in mg/kg[a,b] | |
|---|---|---|
| | Wet-Mass Basis | Dry-Mass Basis |
| Arsenic[c] | 0.796 ± 0.049 | 7.86 ± 0.48 |
| Cadmium[c,d] | 0.155 ± 0.005 | 1.53 ± 0.05 |
| Chromium[c] | 0.233 ± 0.010 | 2.30 ± 0.10 |
| Copper[c,d] | 0.967 ± 0.016 | 9.55 ± 0.16 |
| Iron[e] | 55.1 ± 3.4 | 544 ± 34 |
| Lead[d] | 0.752 ± 0.026 | 7.42 ± 0.26 |
| Nickel[c,d] | 0.109 ± 0.005 | 1.08 ± 0.05 |
| Selenium[c] | 0.224 ± 0.015 | 2.21 ± 0.15 |
| Silver[c,d] | 0.028 ± 0.003 | 0.280 ± 0.033 |
| Tin[d] | 0.028 ± 0.002 | 0.273 ± 0.018 |
| Zinc[c,d] | 12.3 ± 0.3 | 121 ± 3 |

[a] The concentrations are reported on both wet- and dry-mass basis; material as received contains 89.87 % ± 0.05 % (95 % confidence level) water. These elements were determined in freeze-dried samples on a dry-mass basis.

[b] The reference values are the means of results obtained from NRC Canada using one or two analytical techniques and the consensus mean from six laboratories participating in the NRC Canada 14th Intercomparison for Trace Elements in Marine Sediments and Biological Tissues [4]. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [24] with a pooled, within method variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurement [2].

[c] Determined at NRC Canada using GFAAS.

[d] Determined at NRC Canada using ID-ICP-MS.

[e] Determined at NRC Canada using ICP-AES.

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## APPENDIX A

The laboratories listed below performed measurements that contributed to the certification of SRM 1974b Organics in Mussel Tissue (*Mytilus edulis*).

Arthur D. Little, Inc; Cambridge, MA, USA
Australian Nuclear Science and Technology Organization; Menai, NSW, Australia
B & B Laboratories; College Station, TX, USA
BWPC Laboratory; San Francisco, CA, USA
Battelle Pacific Northwest; Sequim, WA, USA
California Department of Fish and Game; Rancho Cordova, CA, USA
City of San Jose Environmental Services Department Laboratory; San Jose, CA, USA
Environment Canada; Moncton, New Brunswick, Canada
Manchester Environmental Laboratory; Port Orchard, WA, USA
NOAA, National Ocean Service, Center for Coastal Environmental Health and Biomolecular Research; Charleston, SC, USA
NOAA, NMFS, Sandy Hook Marine Laboratory; Highlands, NJ, USA
NOAA, NMFS, Northwest Fisheries Science Center; Seattle, WA, USA
Orange County Sanitation District; Fountain Valley, CA, USA
Resource Sciences Centre Department of Natural Resources; Indooroopillly, Queensland, Australia
STL Sacramento; Sacramento, CA, USA
Texas Parks and Wildlife Department; San Marcos, TX, USA
Texas A&M University College of Veterinary Medicine; College Station, TX, USA
University of Connecticut Environmental Research Institute; Storrs, CT, USA
University of Rhode Island Graduate School of Oceanography; Narragansett, RI, USA
US Department of Agriculture, Environmental Chemistry Laboratory; Beltsville, MD, USA
US Geological Survey, National Water Quality Laboratory; Denver, CO, USA
Wright State University; Dayton, OH, USA